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GENERAL DYNAMICS
ASTRONAUTICS

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COMPILATION OF

MATERIALS RESEARCH DATA

Second Summary Report - Phase II

REPORT AE 62-0060-1

For the Period

1 September 1961 to 1 March 1962

GENERAL DYNAMICS/ASTRONAUTICS
San Diego, California
March 1962

Prepared for
Aeronautical Systems Division
Wright-Patterson AFB, Ohio

Contract AF 33(616)-7984, Task No. 73812

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ABSTRACT

This second summary report under Phase II of contract AF 33(616)-7984 contains brief descriptions of materials research projects on which work was in progress during the reporting period at General Dynamics/Astronautics, San Diego, California.

CONTENTS

MATERIALS RESEARCH PROJECT SUMMARY

Period:	1 September 1961 to 1 March 1962
Material Classification:	Plastics and Adhesives
Materials:	Metlbond 406, FM-1000, AF-41, Metlbond 409, Narmco 3170-7131, HT-424, Epon 828/Versamid 125
Title:	An Evaluation of the Mechanical Properties of Adhesives at Cryogenic Temperatures and their Correlation with Molecular Structure
Project No.	REA 111-9206
Objective:	1. To determine the mechanical properties of various adhesives used on several adherends when tested at cryogenic temperatures. Testing will include lap-shear, butt-tensile, impact, and T-peel tests on metal-to-metal bonds. 2. To correlate the chemical compositions and molecular structures of adhesives with properties of their metal-to-metal bonds at cryogenic temperatures.
Results and Conclusions:	Lap-shear tests were run on joints containing 0.020" EFH 301 CRES adherends with both Metlbond 406 and FM-1000 adhesives. In both cases FM-1009-8 tack primer was utilized; results at -423°F were approximately 35% lower than those obtained without primer. Pre-Bond 700 (alkaline cleaner) etching prior to bonding was also evaluated and resulted in higher strengths than all other surface preparation methods. Lap-shear strengths of two new epoxy-nylon systems, tacky AF-41 and Metlbond 409, were also obtained at cryogenic temperatures. A new room temperature curing epoxy-polyamide system filled with finely ground nylon (Narmco 3170-7131) was evaluated with lap-shear specimens utilizing both 0.020" EFH 301 CRES and 0.020" 5Al-2.5Sn titanium alloy adherends. Lap-shear strengths at cryogenic temperatures were poorer than those obtained previously with unfilled epoxy-polyamide systems. Impact specimens were prepared using Metlbond 406, HT-424, FM-1000, and 50:50 Epon 828/Versamid 125. The specimens were tested at 78° and -320°F, and the highest strengths were obtained with the Metlbond 406.

Results and Conclusions: (Continued)

Honeycomb specimens were evaluated in edgewise compression and in pi-tension. The face materials were 0.032" EFH 301 CRES and the core was Hexcel HRP 3/16 (5.5 lb/ft3). Specimens were bonded with FM-1000 and tacky AF-41, and they were tested at -320° and -423°F.

Ultrasonic equipment, initially designed by Diets and co-workers at M.I.T. for the measurement of Young's Modulus of adhesives, has been modified and built. This equipment consists of a driving system to set up longitudinal ultrasonic vibrations in the specimens, a detecting system to indicate the amplitude of vibrations, and a frequency measuring system. This equipment has been checked out by determining the properties of 321 stainless steel and K-Monel.

MATERIALS RESEARCH PROJECT SUMMARY

Period: January 1962 to 1 March 1962

Material Classification: Plastics and Adhesives

Materials: Selection open

Title: Self-Rigidizing and Ultra-Light-Weight Structures

Project No: REA 111-9311

Objective: To develop new and better methods for the erection and self-rigidization of inflatable space structures. It is the aim of the program to develop single layer construction to replace the present double wall structures and to establish a more reliable rigidizing system to replace the present foaming approach. A material is also being considered which could be used in the erection and rigidization phases and subsequently by removed by sublimation.

Results and Conclusions: An extensive literature survey has been conducted to more thoroughly acquaint the researchers on present methods for accomplishing the proposed task. Information was also accumulated on similar programs being conducted by other firms. Considerable data were obtained from previous reports on the effects of high vacuum and high-intensity ultra-violet radiation on polymeric materials.

A laboratory set-up was prepared to determine the effect of these two variables on materials and systems suggested for consideration. Some preliminary data have been obtained.

MATERIALS RESEARCH PROJECT SUMMARY

Period: January 1962 to 1 March 1962

Material Classification: Plastics and adhesives

Materials: Various organic plastics and elastomers

Title: Properties of Organic Materials in Space Environments

Project No: REA 111-9306

Objective:

1) To determine the deleterious effects of space environments on plastics and elastomers.

2) To improve the performance of these materials by modifying composition and structure.

Current Status: A comprehensive literature survey of reports of recent investigations in this field has been initiated.

Plans have been devised for exposing test specimens to high vacuum and ultra-violet light irradiation.

MATERIALS RESEARCH PROJECT SUMMARY

Period: January 1962 to 1 March 1962

Material Classification: Adhesives and Lubricants

Materials: Selection open

Title: Adhesion and Lubrication in Space Environment

Project No: REA 111-9305

Objective:

1) To study the surface and structural variables of a metal that control the adhesion characteristics in a high vacuum.

2) To determine adequate lubrication systems to be used in a high vacuum system.

Current Status:

The design of apparatus for the adhesion and lubrication experiments is underway. Both pieces of equipment will be designed to eliminate de-gassing problems so that hard vacuums can be achieved.

The lubrication test system will incorporate a magnetic clutch drive so that only the test bearing will be exposed to the vacuum. To insure long-time service, the drive motor will be encapsulated so that its bearings will be maintained in an atmosphere.

MATERIALS RESEARCH PROJECT SUMMARY

Period: 1 September 1961 to 1 March 1962

Material Classification: High Temperature Materials

Materials: 301 XFH Stainless Steel, and Titanium Alloys Ti-5Al-2.5Sn and Ti-13V-11Cr-3Al

Title: High Temperature Properties of Selected High Temperature Alloys

Project No: REA 111-9222

Objective:

1. To determine the influence of cold work on the high temperature properties of selected alloys.
2. To determine the effect of cyclic exposure to stress and temperature on the high temperature properties of selected alloys.
3. To determine the influence of melting practice on the high temperature properties of promising high temperature alloys.

Results and Conclusions:

The creep behaviour of the three test materials under cyclic conditions was generally similar to that observed under constant conditions; however, some specific differences were noted.

Type 301 XFH stainless steel had increased creep resistance under cyclic conditions at the higher stresses and longer times, but no difference was noted at lower stresses or shorter times.

Although average creep curves under cyclic and constant conditions appear to be the same for the Ti-5Al-2.5Sn alloy, the large first stage creep observed at 600°F during the first loading cycle did not occur on subsequent loading cycles. Thus, pre-creep could be used as a means of strengthening Ti-5Al-2.5Sn alloy for use at 600°F.

Wide scatter in the cyclic creep data from tests of the Ti-13V-11Cr-3Al alloy prevented clear definition of cyclic creep effects, but the average cyclic creep curves fell below the constant creep curves under all test conditions.

Creep damage (change in room temperature tensile properties after creep testing) from cyclic conditions was similar to that resulting from steady state creep testing.

MATERIALS RESEARCH PROJECT SUMMARY

Period: 1 September 1961 to 1 March 1962

Material Classification: High Temperature Coatings

Materials: AM 355, A-286, Vascojet 1000, K-Monel, Inconel-X, Rene 41, FS 82

Title: Evaluation of High Temperature Protective Coatings for Refractory Metals

Project No: REA 111-9208

Objective:

1) To develop high temperature ceramic baths for heat treating various refractory metals.

2) To evaluate high temperature and oxidation resistant coatings for refractory metals.

Results and Conclusions:

Four of the more promising ceramic baths were selected for further evaluation of harmful effects which might result from their use during the austenitizing of the H-11 die steel. Comparing the hardness values of samples heat treated in these baths and specimens conventionally heat treated in an air atmosphere, no significant differences were found. Microscopic examination disclosed surface cracks in samples subjected to severe (water) quenching rates. However, this effect was clearly independent of the high temperature medium employed, and excellent results were obtained at less severe quenching rates. It is concluded that no detrimental effects should occur from the use of these ceramic heat treat baths.

A thermal gravimetric analysis apparatus was set up to detect weight changes of coated and uncoated FS82 columbium alloy due to oxidation of the metal at elevated temperatures. With this apparatus, the oxidation rates of unprotected samples were determined at temperatures ranging from 1600°F to 2500°F. The rates were found to be parabolic during initial oxidation and linear during the later portion of the test; a definite decrease of the parabolic region was evident as the oxidation temperature was increased. To determine the oxidation mechanism of the FS82 alloy, the inert marker method was used. This test revealed that the oxygen was diffusing inward through the oxide to the metal interface.

Since protective coatings are generally recognized as a requirement for utilization of refractory metals at high temperatures, a survey was made of the commercially available coatings for the FS82 columbium alloy. Four coating manufacturers were asked to apply their product and technique to various tensile, creep and oxidation test specimens. The oxidation test specimens were exposed to a continuous weight gain test of 150 minutes at 1350°, 1800°, 2050°, and 2500°F; additional samples were exposed to ten 15-minute cycles at the same temperatures. Following these tests

Results and Conclusions: the samples were subjected to a 90° angle bend test over a 1.5 t bend radius at room temperature to determine their relative ductility. All of the as received specimens demonstrated complete ductility. However, after 10 cycles at 2500°F the samples from only two companies retained their ductility. At 1800° and 2050°F, samples from one company failed prior to 10 cycles. At 1350°F all of the samples retained their ductility after 10 cycles of test.

MATERIALS RESEARCH PROJECT SUMMARY

Period: 1 September 1961 to 1 March 1962

Material Classification: Materials for Cryogenic Applications

Materials: 301, 304, and 310 stainless steel; 2014-T6, 2219-T81, and 5456-H321 aluminum alloys; and 5Al-2.5Sn Titanium alloy.

Title: Crack Propagation at Cryogenic Temperatures

Project No: REA 111-9302

Objective: To determine the resistance to brittle fracture of several sheet alloys at cryogenic temperatures.

Current Status: The test program includes the determination of K_c and G_c values from 78° to -423°F of parent metal and fusion welded 301, 304 ELC, and 310 stainless steels; 2014-T6, 2219-T81, and 5456-H321 aluminum alloys; and 5Al-2.5Sn titanium alloy.

Modification of the liquid hydrogen cryostat for testing of the 4" wide crack propagation specimens has been completed, and the cryostat for 19" wide specimens is being prepared for testing.

Twenty-two crack propagation specimens have been tested at -423°F during the first two months of this year.

MATERIALS RESEARCH PROJECT SUMMARY

Period:	January 1962 to 1 March 1962
Material Classification:	Materials for cryogenic applications
Materials:	General
Title:	Use of Gettering Agents for Maintaining High Vacua
Project No:	REA 111-9304
Objective:	To enhance and maintain vacua in cryogenic vacuum systems through the use of getter agents.
Current Status:	The existing cryostat is being modified to lessen hydrogen boil-off. A literature search has been conducted. Materials selected as getter agents include molecular sieves, palladium-coated alumina catalysts, activated carbon, and silica gels.

MATERIALS RESEARCH PROJECT SUMMARY

Period: January 1962 to 1 March 1962

Material Classification: Physical Properties of Metals and Non-Metals

Materials: Selection open

Title: Thermal Expansion of Space Vehicle Materials

Project No: 1471-9303

Objective: To investigate low temperature thermal expansion characteristics of plastics, ceramics, and metals which show promise of use in future space vehicles.

Current Status: A cryostat originally used for low temperature tensile testing has been rebuilt to provide a low boil-off liquid hydrogen bath for the quartz tube dilatometer.

The low temperature furnace for the Leitz dilatometer was modified to provide an interior heater instead of an exterior heater. Several trial runs indicated a power requirement 60% less than that which was necessary with the original winding. This will also result in saving approximately 60 liters of liquid nitrogen per run.

MATERIALS RESEARCH PROJECT SUMMARY

Period: 1 September 1961 to 1 March 1962

Material Classification: Insulation Materials

Materials: Selection open

Title: Advanced Thermal Insulation Systems for Aerospace Vehicles

Project No: REA 111-9310

Objective:

1) To complete the modification and check-out of the test apparatus

2) To establish a test program which will most effectively utilize the capabilities of this equipment.

Current Status:

This apparatus was designed (1) to simulate a cryogenically fueled vehicle protected by systems capable of withstanding 1800°F on the hot side and (2) to evaluate these systems for thermal performance at pressures low enough to take advantage of vacuum ("super") insulation.

Construction of the device was completed in July 1961, and performance tests were conducted throughout the latter half of the year.

Thus far in 1962 the data obtained from operation of the equipment have been analyzed, and the necessary modifications to the facility have been initiated. Research is being conducted to determine the most promising areas of investigation; test panels and programs are being planned accordingly.